Original Article

A comparative evaluation of the effect of simulated porcelain firing cycle on the mechanical properties and microstructure of nickel-chromium-based ceramic alloys

Sanjay B. Lagdive, Suresh Meshram[1]
Department of Prosthetic Dentistry, Rural Dental College and Hospital, Pravara Medical Trust,
Loni-412 736, [1]Government Dental College and Hospital, Mumbai, India

For correspondence
Dr. Sanjay B. Lagdive, Department of Prosthetic Dentistry, Rural Dental College, Pravara Rural University, Loni, India.
E-mail: lagdive_san@yahoo.co.in

The present study was carried out to evaluate the effect of porcelain firing cycle on the mechanical properties and microstructure of nickel-chromium-based alloys. Four different alloys were used: Supercast-np, d-Sign 10, Remanium CSe and Maxibond EMCS.

Tensile bars and disks were prepared for testing according to specifications. For each alloy, ten tensile bars and six disks were prepared. For each alloy, five tensile bars and three disks were randomly selected for heat treatment. For heat treatment, the specimens were subjected to simulated porcelain firing cycle in a ceramic furnace.

Both as-cast and heat-treated tensile bars were tested on a universal testing machine, and the values for the ultimate tensile strength, 0.2% offset yield strength and percentage elongations were obtained. Hardness was measured using Vickers's hardness tester. Microstructures were evaluated under an optical microscope.

The data collected were tabulated and subjected to statistical analyses.

Key words: Microstructure, remanium, simulated

INTRODUCTION

It is the dream of a prosthodontist to achieve excellence in the form, function and esthetics to provide life-like restorations. The practice of fixed prosthodontics has been continually evolving for achieving this objective.

Various materials have been used for the restoration of teeth. Metals such as gold, silver and their alloys; base metal alloys; and tooth-colored nonmetallic materials such as ceramics, resins and composites have dominated the field of fixed prosthodontics for many years.

Presently, the ceramometal restorations are widely used since they combine the requirements of strength with metal and that of the esthetic value with ceramic.[1] Alloys used for the construction of porcelain-veneer crowns should fulfill other requirements. These alloys should match the porcelain with regard to the coefficient of thermal expansion; in addition, they should exhibit mechanical properties such as strength, hardness and high temperature strength.[2,3] Among the various metal alloys, large differences exist in their physical properties and handling; thus, considerable research has been directed toward the investigation of properties of these alloys.

Of considerable interest has been the effect of firing cycles on the alloys. This information is required because the firing porcelain in the metal substructure of a restoration may cause changes in the mechanical properties and microstructure, which can influence the behavior of alloys on prolonged use.[4]

Hence, in this study, the effect of simulated porcelain firing cycle on the mechanical properties and microstructure of nickel-chromium-based ceramic alloys was evaluated.

Aims and objectives

1. To evaluate and compare the mechanical properties of nickel-chromium-based alloys.
2. To evaluate and compare the effect of heat treatment on the mechanical properties of nickel-chromium-based ceramic alloys.
3. To evaluate the effect of heat treatment on the microstructure of nickel-chromium-based ceramic alloys.

Review of literature

Considerable research has been directed toward the investigation of the properties of nickel-chromium-based alloys. These studies include casting accuracy, corrosion–resistance, castability of the metal and the effect of heat treatment.

Moffa *et al*[5] advocated that values for hardness, rigidity, resistance to permanent deformation, sag resistance and bond strength of the non-precious alloys to porcelain were significantly greater than those of the gold-based alloys.

Huget *et al*[6] evaluated the properties of two nickel-chromium crown-and-bridge alloys, namely, Microbond-NP and Wiron-S. Microbond-NP causes a reduction in strength and an increase in the length. The properties of Wiron-S were not significantly altered by the heat treatment.

Morris, a participant of CSP No. 147 / 242[4] concluded that the effects of heat treatment on the strength of the palladium-based alloys were variable, showing a decrease in the hardness and an increase in the length. The nickel-chromium-based alloys were weakened by heat treatment and showed a decrease in hardness and increase in length. Heat treatment did not significantly affect the modulus of elasticity in any of the tested alloys.

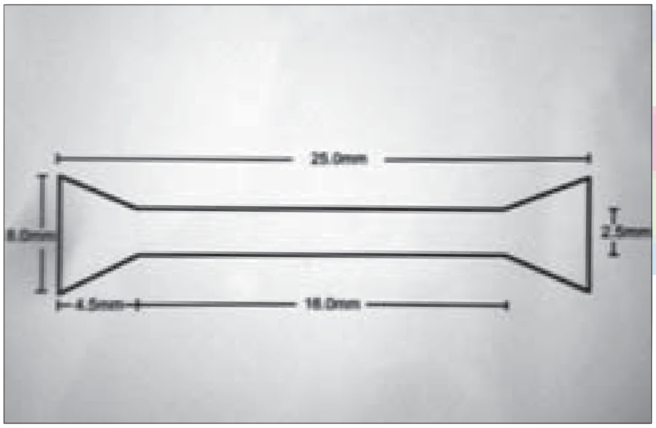

Figure 1: Dimension and design of the cast tensile test specimens as described by Nicholls and Lemm (1985)

MATERIALS AND METHODS

Four types of nickel-chromium-based ceramic alloys having different compositions were selected; these alloys are more commonly used and easily available. The following are the alloys selected:

- Gr. A: Supercast-np {Thermobond alloy, mfg. USA}
- Gr. B: d-Sign 10 {Williams, USA}
- Gr. C: Remanium CSe {Dentaurum, GERMANY}
- Gr. D: Maxibond EMCS {Thermobond alloy, mfg. USA}

Preparation of cast specimens

Two types of specimen were prepared:

1. Tensile bars: Tensile bars were prepared as described by Nicholl's and Lemm,[1,7] and their dimensions are shown below [Figure 1].

   Tensile bars were used for the testing of the following features:
   - Ultimate tensile strength
   - 0.2% offset yield strength
   - Percent elongation

   Tensile bars were prepared by the injection of molten wax into split metal molds to ensure consistent dimensions.
2. Disks: Disk specimens were 13 mm in diameter and 3 mm in thickness; these were prepared by the injection of molten wax into metal molds.[6–9]

   These disks were used for testing of the following features:
   - Hardness
   - Microstructure evaluation

   A total of 40 tensile bars and 24 disks were prepared using wax. All specimens were then allotted to four groups. For each alloy, 10 tensile bars and 6 disks were selected. The patterns were invested

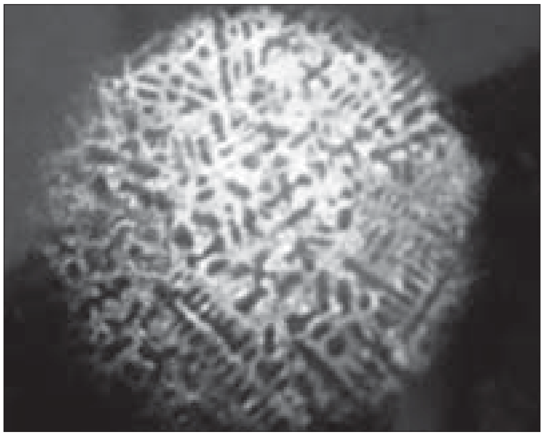

Figure 2: Microstructure of group B alloy in as-cast condition at 200×

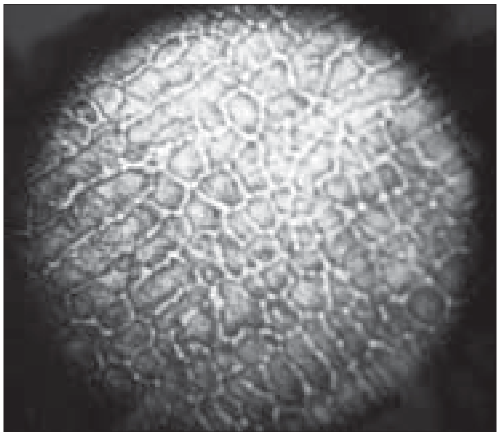

Figure 3: Microstructure of group B alloy in heat-treated condition at 200×

using phosphate-bonded investing material. After bench set, burnout was performed according to the instructions and cast with respective alloys.

Heat treatment [H/T]

For each alloy, five tensile bars and three cast disk specimens were randomly selected and subjected to heat treatment. The alloy specimens were heat treated by the time-temperature sequence used in the porcelain firing cycle in a ceramic furnace, but without the application of porcelain, as shown in Table 1.

Heat treatment involved the following four steps:

1. Degassing: Degassing was followed for each alloy according to manufacturers' instructions.
2. Simulated opaque porcelain firing
   i) 1st opaque firing
   ii) 2nd opaque firing
3. Simulated body porcelain firing
   i) 1st body firing
   ii) 2nd body firing
4. Simulated firing for glaze

Testing of specimens

All tensile bars were tested on a universal testing machine at a crosshead speed of 0.02 inches per minute. Special metal grips were constructed to hold the specimens. A strain gauge extensometer was used to record the resulting strain, and elongation measured was more than 12.5 mm gauge length. For each tensile bar, the value of ultimate tensile strength, 0.2% offset yield strength and percent elongation, were determined.

Testing of hardness and microstructure

For evaluating hardness and microstructure, the disks were plastic mounted. The disks were then sequentially polished with 1/0, 2/0, 3/0 and 4/0 grit emery abrasive papers, diamond paste (6 µm) and fine alumina abrasive (0.05 m).

Hardness was measured by Vickers's hardness tester equipped with a diamond-indenting tool, with an apex angle of 120° under a load of 10 kg.

Specimens for metallographic examination were etched by immersing in a solution containing 92% hydrochloric acid, 5% sulfuric acid and 3% nitric acid by volume for 1.5 min. The specimens were then observed with an optical microscope.

RESULTS

The data collected were subjected to statistical

Table 1: Simulated porcelain firing cycle

Ceramic firing program	Holding temp. (T)	Temp. increase (C/min)	Standby temp (°C)	Holding time (Min)	Vacuum on (°C)	Vacuum off (°C)
Oxidation for A, C and D Gr. alloy	980	30	550	5	0	0
For B Gr.	1010	30	550	5	0	0
1st opaque	980	80	403	1	550	1 below T
2nd opaque	970	80	403	1	550	1 below T
1st body	920	60	403	1	580	1 below T
2nd body	910	60	403	1	580	1 below T
Glaze	900	60	403	1	0	0

Table 2: Descriptive statistics

Mechanical properties	As-cast				After heat treatment				
	Sub groups	Mean	S.D.	S.E. Mean	Sub group	Mean	S.D.	S.E. Mean	*P* Value
Ultimate tensile strength (Mpa)	A	941	65.92	29.48	A1	817.2	144.35	69.56	0.019
	B	958	60	26.83	B1	627	41.47	18.55	0.000
	C	511	50.2	22.45	C1	459	31.62	14.14	0.086
	D	575	29.66	13.27	D1	479	126.49	56.57	0.137
0.2% offset yield strength (Mpa)	A	543	63.87	28.57	A1	375	129.15	57.76	0.310
	B	675	79.56	35.58	B1	479.2	13.79	6.17	0.001
	C	387	41.47	18.55	C1	299	81.24	36.33	0.063
	D	383.2	35.61	15.93	D1	293	79.25	35.44	0.049
Percent elongation (%)	A	7.8	6.76	3.02	A1	12.8	2.39	1.07	0.158
	B	12.6	4.39	1.96	B1	22.8	11.43	5.11	0.100
	C	15.6	6.23	2.79	C1	17.6	4.93	2.20	0.589
	D	13.4	2.51	1.12	D1	26	11.2	5.01	0.040
Vicker's hardness (VPN)	A	420	6	3.46	A1	202	6.39	4.00	0.000
	B	416.3	5.03	2.91	B1	222	5.57	3.21	0.000
	C	218.7	1.3.01	7.51	C1	191.7	4.51	2.60	0.027
	D	211	5.57	3.21	D1	205.3	4.04	2.33	0.227

$P < 0.05$, significant difference

analysis. The means of the alloys within the as-cast and heat-treated samples were analyzed for differences by using the analysis of variance (ANOVA).

Paired comparison of the alloys in the as-cast and heat-treated samples was analyzed by applying Tukey's HSD multiple comparison test. *t*-Test was used to analyze differences in means of the alloys in the as-cast and heat-treated conditions.

Mechanical properties of as-cast alloys [Table 2]:

- Ultimate tensile strength: The means ranged from 958 MPa for d-Sign 10 to 511 MPa for Remanium CSe.
- 0.2% offset yield strength: The means ranged from 675 MPa for d-Sign 10 to 383 MPa for Maxibond EMCS.
- Percent elongation: Remanium Cse (15.6%) exhibited the highest percent elongation and Supercast-np exhibited the lowest mean percent elongation (7.8%).
- Vickers's hardness: The means ranged from 420 VPN for Supercast-np to 211 VPN for Maxibond EMCS.

Effect of porcelain firing cycle on mechanical properties of the alloys [Table 2]:

- Ultimate tensile strength: The means ranged from 817.2 MPa for Supercast-np to 459 MPa for Remanium CSe. Supercast-np and d-Sign 10 showed significant differences in the values after heat treatment; the other two alloys showed no significant difference in the values.
- 0.2% offset yield strength: The means ranged from 479.2 MPa for d-Sign 10 to 293 MPa for Maxibond EMCS. There was a decrease in the strength for all alloys, although Remanium CSe and Maxibond EMCS showed no significant differences in the values after heat treatment.
- Percent elongation: Percent elongation increased for all alloys. The highest percentage was recorded for Maxibond EMCS (26%) and the lowest, for Supercast-np (12.8%). Only Maxibond EMCS exhibited significantly high percent elongation after heat treatment.
- Vickers's hardness: The highest hardness was exhibited by d-Sign 10 (222 VPN) and the lowest, by Remanium CSe (191.67 VPN). A significant decrease occurred in the hardness of each alloy; only Maxibond EMCS did not show any significant decrease.

Microstructure evaluation

- Supercast-np:

  As-cast exhibited highly segregated coarse dendrites in the gamma matrix [Figure 2].

  After H/T, fully homogenized polygonal grains of the gamma phase with slightly thick grain boundaries were observed [Figure 3].
- d-Sign 10:

  As-cast showed fine dendrites of the gamma phase with interdendritic matrix.

  After H/T, homogenized polygonal coarse grains with thick, surrounding boundaries were observed; few precipitates of nickel aluminate were visible inside the grains.
- Remanium CSe

  As-cast showed fine dendritic segregation.

  After H/T, an uneven structure and gamma prime precipitates were observed in the gamma phase matrix. Homogenization was observed in the microstructure.
- Maxibond EMCS

  As-cast fine dendritic segregation was the same as that observed for Remanium Cse.

  After H/T, no appreciable change was observed in the microstructure except for an increase in the grain size.

DISCUSSION

All the nickel-based alloys became weak after heat treatment and exhibited a decrease in the ultimate tensile strength, 0.2% offset yield strength, hardness and increase in percent elongation. These results agree with those of the previous studies carried out by Moffa *et al*,[5] Huget,[6,10] Morris *et al*[4] and Chew *et al*.[1]

Differences in mechanical properties exhibited by the alloys were associated with the differences in their composition and microstructure.

All alloys were primarily strengthened by solid solution hardening. Precipitation played a major role in the hardening of the alloys. However, these alloys were not hardened by precipitation of similar composition.

In as-cast condition, all the alloys exhibited a dendritic structure, i.e., nonhomogeneous structure, which is responsible for the higher strength of the alloys.

After heat treatment, the microstructure of the alloys showed improvement in dendritic structure, i.e., homogenization, which results in good grain refinement.[11] Hence, the improvement in ductility and concurrent reduction in strength was observed. After heat treatment, d-Sign 10 exhibited the highest 0.2% yield strength. This might be due to the high aluminum content (3.3%) of the alloys.

The microstructure of Maxibond EMCS after heat treatment exhibited little change; this finding supports the slight change in the mechanical properties of the alloy.

The Supercast-np and d-Sign 10 can permit the use of thinner castings and greater thickness of porcelain veneering material. Higher strengths of these alloys support their use in the long-term fixed partial dentures. Restorations performed using these alloys are more likely to abrade the opposing dentition and are difficult to polish.

The low strength of Remanium CSe and Maxibond EMCS, contribute to easy burnishability and easy finishing.

The properties of the ultimate tensile strength, 0.2% offset yield strength, percent elongation and hardness are not the only parameters determining clinical success. The interaction between these and other parameters such as tarnish-corrosion resistance, porcelain-metal compatibility and biocompatibility, which were not included in this study, should also be considered when judging the clinical success of the restorations.

CONCLUSIONS

Considering the mechanical properties of the alloys, following conclusions can be drawn:

1. Based on the ultimate tensile strength, the alloys can be arranged in the following order: d-Sign 10 (958 Mpa) > Supercast-np (941 Mpa) > Maxibond EMCS (575 Mpa) > Remanium Cse (511 Mpa).
2. According to 0.2% offset yield strength, the alloys can be arranged in the following sequence: d-Sign 10 (675 Mpa) > Supercast-np (543 Mpa) > Remanium Cse (387 Mpa) > Maxibond EMCS (383.2 Mpa).
3. According to percent elongation, the alloys can be arranged in the following order: Supercast-np (7.8%) > d-Sign 10 (12.6%) > Maxibond EMCS (13.4%) > Remanium Cse (15.6%).
4. According to Vickers's hardness, the alloys can be arranged in the following sequence: Supercast-np (420 VPN) > d-Sign 10 (416.33 VPN) > Remanium CSe (218.67 VPN) > Maxibond EMCS (211 VPN).
5. After heat treatment, all the nickel-based alloys became weak.
6. For all alloys, the ultimate tensile strength decreased after heat treatment; these alloys can be as arranged in the following sequence: Supercast-np (817.2 MPa) > d-Sign 10 (627 MPa) > Maxibond EMCS (479 MPa) > Remanium CSe (459 MPa).
7. After heat treatment, 0.2% offset yield strength showed a decrease in the values; they can be arranged in the following ordered: d-Sign 10 (479.2 MPa) > Supercast-np (375 MPa) > Remanium CSe (299 MPa) > Maxibond EMCS (293 MPa).
8. After heat treatment, percent elongation showed an increase in the values; the alloys can be arranged in the following order: Supercast-np (12.8%) > Remanium CSe (17.6%) > d-Sign 10 (22.6%) > Maxibond EMCS (26%).
9. The hardness values for all alloys decreased after heat treatment; the alloys can be as arranged in the following sequence: d-Sign 10 (222 VPN) > Maxibond EMCS (205.33 VPN) > Supercast-np (202 VPN) > Remanium CSe (191.67 VPN).
10. Microstructure evaluation of the alloys in as-cast condition showed dendritic structure. After heat treatment, all the alloys exhibited homogenization, which resulted in good grain refinement.

REFERENCES

1. Chew CL, Norman RD, Steward GP. Mechanical properties of metal ceramic alloys at higher temperature. Dent Mater 1990;6:223-7.
2. Hobo S, Shillingburg HT. Porcelain- fused- to-metal tooth preparation and coping design, J Prosthet Dent 1973;30:28-36.
3. O' Brien WJ, Kring JE, Ryge G. Heat treatment of the alloys to be used for the fused porcelain technique, J Prosthet Dent 1964;14:955-60.
4. Morris HF, Participants of CSP No. 147/242: Veterans administration cooperative studies project No. 147/242 Part VII: The mechanical properties of metal ceramic alloys as cast and after simulated porcelain firing. J Prosthet Dent 1989;61:160-9.
5. Moffa JP, Lugassy AA, Guckes AD, Gettleman L. An evaluation of non-precious alloys for use with porcelain veneers. Part I - Physical properties, J Prosthet Dent 1973;30:424-31.
6. Huget EF, Dvivedi N, Cosner H. Properties of two nickel chromium crown and bridge alloys for porcelain veneering, J Am Dent Assoc 1977;94:87-90.
7. Nicholls JI, Lemm RW. Tensile strength of presoldered and post soldered joints. J Prosthet Dent 1985;53: 476-82.
8. Vermilyea SG, Huget EF, Vlica JM. Observations on gold-palladium-silver and gold-palladium alloys. J Prosthet Dent 1980;44:294-9.
9. Vermilyea SG, Mills DE: Observations on nickel-free, beryllium free alloys for fixed prosthesis, J Am Dent Assoc 1983;106:36-8.
10. Huget EF, Vlica JM, Wall RM. Characterization of two ceramic base metal alloys. J Prosthet Dent 1978;40: 637-41.
11. ASM Handbook. Metallographic and microstructure, 9th ed. USA: ASM Int, the Materials Information Soc; 1990. p. 340-1.

Source of Support: Nil, **Conflict of Interest:** None declared.